Journal of Food Engineering 111 (2012) 432–439

Contents lists available at SciVerse ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Clarification of passion fruit juice by microfiltration: Analyses of operating parameters, study of membrane fouling and juice quality

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ARTICLE INFO

Article history:
Received 23 August 2011
Received in revised form 8 January 2012
Accepted 28 January 2012
Available online 16 February 2012

Keywords:
Passion fruit
Microfiltration
Fouling mechanism
Bayesian inference

ABSTRACT

In this study, the performance of two membranes were compared – tubular ceramic and hollow fiber poly(imide) – under transmembrane pressure of 0.5 and 1 bar, for the clarification of passion fruit pulp pre-treated by centrifugation and enzymatic treatment at the concentrations of 150 and 300 ppm. Nutritional and sensorial qualities of the clarified juice obtained were evaluated. Thus, it was possible to observe that the most adequate condition for the clarification of passion fruit pulp was with enzymatic treatment at 150 ppm and its posterior microfiltration at the ceramic tubular membrane of 0.3 μm with transmembrane pressure of 0.5 bar. The fouling mechanism was identified by estimation of model parameters according to a nonlinear regression by Bayesian inference. Analysis of the fouling mechanism results revealed that hollow fiber membrane is controlled by a cake filtration mechanism, and internal pore blocking fouling mechanism controls ceramic tubular membrane.

1. Introduction

Nowadays, there is a worldwide increasing tendency for the consumption of tropical fruits, juices and fruit drinks, due to the consumer interest in healthy products which are practical and ready to be consumed. Passion fruit (*Passiflora edulis f. flavicarpa*) juice is globally marketed, mainly for its flavor and unique aroma. Brazil, Ecuador and Colombia are the biggest passion fruit producers in the world. According to Jiraratananon and Chanachai (1996), the concentration process for passion fruit juice employs flash evaporation, which is still a common process for fruit juice concentration. Therefore, the development and study of new technologies to obtain high quality products from the fruit is essential to encourage its production.

Membrane processes are systems used in various production sectors today, since this separation process is athermal and involves no phase change or chemical agents. The introduction of these technologies in the manufacture of fruit juices represents one of the technological answers to the problem of producing an additive-free juice which has high quality, and natural fresh taste. Juice clarification, stabilization, depectinization and concentration are typical steps in which membrane processes such as microfiltration, ultrafiltration, nanofiltration and reverse osmosis have been successfully utilized (Fukomoto et al., 1998; Gokmen et al., 1998; Vaillant et al., 2001; Cassano et al., 2007). Similarly, clarifications based on membrane processes, particularly ultrafiltration and microfiltration, have replaced conventional fining, resulting in the elimination of the use of fining agents and a simplified process for continuous production.

Hollow fiber membranes have become ubiquitous in many fields such as waste water treatment (Yeo et al., 2006; Albasi et al., 2002; Busch et al., 2007), biomedical engineering (Bartolo et al., 2009) and processing of beverages (Girard and Fukumoto, 2000) because of their low manufacturing costs and simple handling. Moreover, one of the main advantages of this filtration configuration is the high membrane area per volume unit of module in comparison with other configurations of membranes.

One of the biggest problems of using membrane for clarifying juices is the decay of permeate flux, which is caused by the phenomenon known as fouling (Cassano et al., 2007; Barros et al., 2003; Ushikubo et al., 2007). To become a feasible technique, the membrane process has to achieve acceptable values of permeate flux. Studies indicate that enzymatic treatment helps to increase permeate flux, given that insoluble pectic, cellulosic materials and polygalacturonase are the main foulants (Vaillant et al., 2001, 1999; Cassano et al., 2007; Barros et al., 2003; Ushikubo et al., 2007; Campos et al., 2002; Matta et al., 2000; Oliveira et al., 2010; Rai et al., 2007; Watanabe et al., 2006). Apart from enzymatic treatment, juice centrifugation is a common pre-treatment method (Oliveira et al., 2010; Rai et al., 2007). Several studies indicate that transmembrane pressure increase can lead to a positive effect in flux, since it is the driving force (Jiraratananon and Chanachai, 1996; Ushikubo et al., 2007; Oliveira et al., 2010). On the other hand, fouling and polarized layer are more accentuated under higher transmembrane pressure in passion fruit juice clarification.

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0260-8774
doi:10.1016/j.jfoodeng.2012.01.021

Nomenclature

J_t	Flux at any time (kg h^{-1} m^{-2})	A	Membrane area (m^2)
J_0	Initial flux (kg h^{-1} m^{-2})	R_M	Intrinsic membrane resistance (m^{-1})
t	Time (s)	ε	Membrane porosity (m)
b_1, b_2, b_3, b_4, b_5	Constant that characterizes fouling mechanism (unit depends on mechanism)	λ	Membrane thickness (m)

The aims of this work were: (i) to compare microfiltration by ceramic tubular and poly(imide) hollow fiber membranes for the clarification of pre-treated passion fruit pulp, trying to obtain a clarified, sterile and integral product, and (ii) to study the fouling mechanism by Bayesian inference.

2. Theory

2.1. Mathematical models of fouling mechanism

Various modes of pore blocking are a function of the solid/solute size and shape in relation to the membrane pore size distribution: (i) complete pore blocking - the pore entrance is sealed; (ii) pore bridging - partial obstruction of the entrance; (iii) internal pore blinding - material not rejected by the pore entrance is adsorbed or trapped on the pore wall or in the membrane support (Barros et al., 2003). For engineering processes, designing systems may be useful to classify fouling as in-depth pore fouling, pore plugging and cake formation. Modes are illustrated in Fig. 1.

According to Cheryan and Alvarez (1995), several models relate the flux to the time or permeated volume and generally take an exponential form, considering the shapes of fouling curves. Eqs. (1) and (2) are some examples,

$$J_t = J_0 t^{-b_1} \tag{1}$$

$$J_t = J_0 e^{-b_2 t} \tag{2}$$

In the above equations, J_0 is the initial flux; J_t is the flux at any time t; and b_1 and b_2 are constants characterizing the fouling processes.

According to literature, several models have been developed based on pore blocking mechanisms developed for dead-end filtration (Barros et al., 2003; Field et al., 1996; Hérmia, 1982. When particles are larger than pore size, membrane portion of the filtration area reached by particles is blocked as a consequence of a complete pore obstruction by means of sealing–blocking (Barros et al., 2003). Cheryan and Alvarez (1995) affirm that the model takes the form of Eq. (3) in this case of pore blocking.

$$J_t = J_0 e^{-J_0 b_3 t} \tag{3}$$

Cake filtration occurs when particles or macromolecules that do not enter the pores, form a cake on the membrane surface. The overall resistance is composed of a cake resistance and a membrane resistance, which is assumed to remain unchanged (Barros et al., 2003). According to Cheryan and Alvarez (1995), the model takes the form of Eq. (4).

$$J_t = J_0 \left(1 + \frac{2AJ_0 b_4 t}{R_M}\right)^{-\frac{1}{2}} \tag{4}$$

Here A is the membrane area and R_M is the intrinsic membrane resistance.

When particles enter the pores and either get deposited or adsorbed to reduce the pore volume, internal pore blocking occurs. The irregularity of pore passages causes the particle to become tightly fixed blinding the pore. In this case, membrane resistance increases as a consequence of pore size reduction. Besides, if internal pore blocking occurs, fouling becomes independent of cross-flow speed and no limiting value for the flux may be attained. According to Cheryan and Alvarez (1995), the model takes the form of Eq. (5).

$$J_t = J_0 \left(1 + \frac{J_0 b_5 t}{\varepsilon \lambda}\right)^{-2} \tag{5}$$

where λ is the membrane thickness and ε is the membrane porosity.

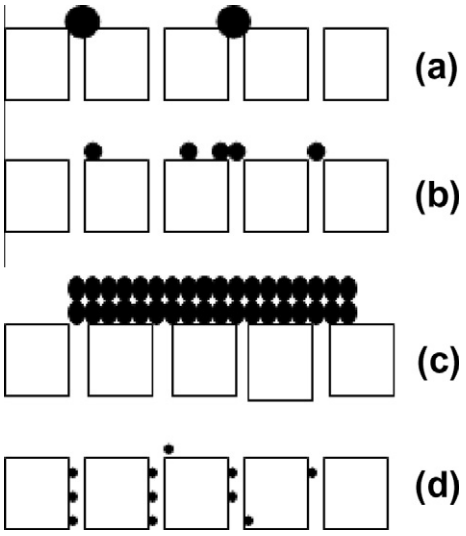

Fig. 1. Mechanism for membrane fouling: (a) complete pore blocking, (b) partial pore blocking, (c) cake filtration, (d) internal pore blocking.

2.2. Bayesian inference

In general, the study of fouling mechanism models evaluation has been carried out by a frequentist approach adjusting nonlinear models, which aim to synthesize pieces of information into parameter estimates to be interpreted. The estimation is based on iterative processes such as that of Gauss–Newton, DUD and Marquardt algorithm, due to the nonlinearity of variables. Such procedures minimize the sum of residue squares. However, when individual adjustments are considered, i.e. adjustments for many experimental units of mathematically complex models or there are few possible longitudinal observations, interactive methods frequently provide negative estimates for parameters. This can cause the formation of atypical curves.

Furthermore, regarding comparisons of curves derived from different treatments, the distribution of nonlinear model parameter estimators do not usually follow Gaussian distribution. Therefore, the process to formulate statistical tests becomes complex once presuppositions related to the asymptotic theory are not attended.

In recent studies involving the adjustment of nonlinear regression models, Bayesian inference was successfully used, as it

reduced the number of biased estimations even when little information was used.

3. Materials and methods

3.1. Pulp fruit and pre-treatments

The passion fruit pulp used in this work was supplied by PolpaNorte (Japurá, Paraná, Brazil), and was kept under refrigeration of −8 °C until being processed. Before microfiltration passion fruit pulp was centrifuged to try to minimize fouling. Passion fruit whole pulp centrifugation was carried out in a rotor centrifuge (Chibas Center), with a system made of stainless steel 316 treatment/feeding tank, rotor with stainless steel 316 internal parts (rotor and cover), carbon steel carcass and a stainless steel 304 collector tank, presenting switching with electric engine of 5 CV linked to a pulley, providing 17,000 rpm and a lung tank with 150 L of capacity.

After centrifugation, the fruit pulp was treated with Pectinex Ultra SP-L (Novozymes, Denmark) at concentrations of 150 and 300 ppm at hydrolysis temperature of 50 °C for an hour under constant shaking.

3.2. Microfiltered pilot plant description and membranes

Clarification experiments were carried out in the laboratorial microfiltration unit, whose scheme is shown in Fig. 2. On the laboratory microfiltration unit, it was possible to use different filtration modules. The first was made of stainless steel, containing a ceramic tubular membrane (material: α-Al_2O_3/TiO_2, mean pore = 0.3 μm, internal diameter = 7 mm, area = 0.005 m^2). The second module consisted of polyamide hollow fiber membrane from PAM Membranas Seletivas LTDA (mean pore = 0.3 μm, external diameter = 0.8–1.0 mm, area = 0.0158 m^2). These modules were connected through tri-clamp connections.

The unit also contained a feed tank (T1) with 5 L capacity, a pump (P1), a flowmeter (F1), two pressures gauges (PG1, PG2), a thermometer (T2), and six gauges (V1–V6). The adjustment of operating conditions was done by controlling pump valves and engine rotation simultaneously. Juice temperature was maintained through water circulation in the tank jacket (C1).

3.3. Microfiltration procedure

Diluted pulp (3.0 L) was place into feed tank and was circulated in the microfiltration unit. Feed stream was pumped from a temperature controller tank (25 °C) through the membrane; flow rates were 500 L/h (Re = 10,800) and 325 L/h (Re = 250) for ceramic tubular membrane and polyamide hollow fiber membrane, respectively. The hollow fiber membrane recirculation fluid dynamics was, at first, also established at 500 L/h. However, it caused the fibers rupture and, for this reason, a fluid dynamics of 325 L/h, the highest for the microfiltration membrane, was adopted. Transmembrane pressure was 0.5 and 1.0×10^5 Pa.

Permeate was collected in a beaker placed on an electronic balance (±0.01 g) (Gehaka, São Paulo, Brazil) and the concentrate returned to the feed tank. In this way, permeate mass was controlled and concentration factor (CF) defined by Eq. (6). When CF reached the value of three, the experiment was finished.

$$CF = \frac{V_f}{V_f - V_p} \tag{6}$$

in which V_f and V_p are the total feed volume and permeate volume, respectively. Permeate flux was calculated according to Eq. (7), and curve J versus t was obtained for each run.

$$J = \frac{m_p}{A.t} \tag{7}$$

In the above equation, m_p is the permeate mass; A is the effective membrane area; and t is the time. To obtain flux (J) in terms of volume, the expression for J (Eq. (7)) was divided by passion fruit juice density.

3.4. Physico-chemical analysis

Feed samples, concentrate and permeate were analyzed: for pH at 25 °C (using a Digimed DM 20 pH meter), soluble solids at 20 °C (measured with a Shimadzu refractometer, Japan), turbidity (measured with Hach DR/2010 Datalogging Spectrophotometer), color and absorbance (measured with Hach DR/2010 Datalogging Spectrophotometer), reducing sugars, total sugars (AOAC, 1984), ascorbic acid (Adolfo Lutz, 1985), and galacturonic acid (Kintner and van Buren, 1982). Physical and chemical analyses of samples

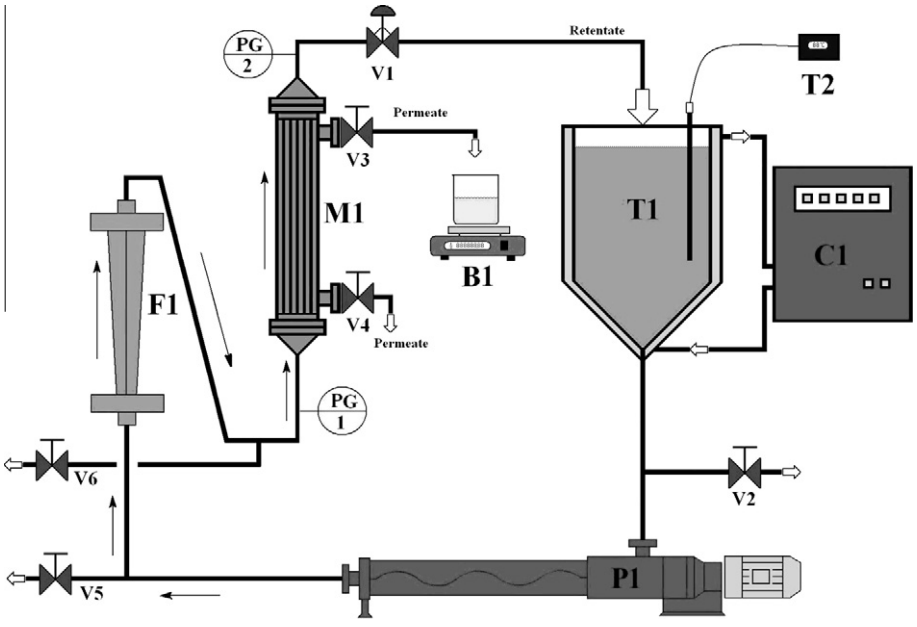

Fig. 2. Schematic diagram of laboratory microfiltration unit.

of permeate, concentrate and feed were undertaken in triplicate. Results expressed are the arithmetic averages of values found.

3.5. Microbiological analyses

The laboratorial microfiltration unit was sterilized by alcohol (70%). Membranes and beakers were sterilized by a solution of hypochlorite at 1% for 30 min.

Microbiological analyses-counting of coliform bacteria, *salmonella* sp mould and yeast, and standard counting in mesopohile dish of the permeate juice were carried out in the best condition of permeate flux. These analyses were carried out according to the methodology of Siqueira (1995).

3.6. Sensorial analysis

For the analysis, juice of these samples was prepared by diluting the products in two parts of water, and then they were equally sweetened. The juices were served in small disposable neutral plastic cups, coded with three-digit numbers and disposed randomly together with a glass of water for palate washing.

The test was applied to 32 non-tested tasters. They received three samples of about 50 mL, at a temperature of 10 °C. Samples were labeled as the following: the first sample was passion fruit whole pulp juice (without microfiltration); the second one was juice from the hollow fiber membrane permeate; the third sample was juice from the tubular membrane permeate. The last ones were at the best conditions found in this study after physical and chemical evaluations.

It was required that the tasters evaluated their acceptance in relation to flavor, having as supporting scale of nine points, varying from "I really liked it" to "I really disliked it", and wrote their opinion down on the opinion form (Monteiro, 2005).

As environmental conditions for the test, blue-lighted individual cabins were established. Data obtained were statistically treated by the variance analyses through the analysis tool ANOVA (using software EXCEL® 2010), at level of 95% by Tukey's test, determining the evaluation result.

3.7. Fouling mechanism study

For the first stage it was assumed that the fluxes (J_t) have normal distribution, i.e.:

$$J_t \sim Normal(\mu, \sigma)$$

The models are shown in Eqs. (1)–(5), in which Eqs. (1) and (2) relate the flux to the time, and Eqs. (3)–(5) describe fouling mechanism. All model parameters were considered, prior, as non informative Gamma distribution, i.e.:

$$parameters \sim gamma(10^{-3}, 10^{-3})$$

Posterior distributions of parameters were obtained by BRugs on R program (R Development Core Team). 2000,000 samples were obtained by Monte Carlo Markov Chain (MCMC), from which 1000 were discarded ("burn-in samples") to eliminate the effect of initial values. Final samples were taken with steps of one which contain 1999,000 obtained values. Convergence chains were verified by Convergence Diagnosis and Output Analysis - CODA program by Geweke (1992) and Heidelberger and Welch (1983) criteria.

The DIC (deviance information criterion) was used as comparison as well the selection of (co)variables in models. Spigelhalter et al. (2002) suggest the use of difference criterion module between values of DICs of two models, A and B analyzed. This criterion is shown in Eq. (8).

$$D = |\mathrm{DIC_A} - DIC_B| \quad (8)$$

If $D < 5$, there is no significant difference; if $5 \leqslant D \leqslant 10$, there is significant difference; and if $D > 10$, there is highly significant difference.

4. Results and discussion

4.1. Microfiltration performance

The centrifuged passion fruit pulp produced 94% (in mass) of pre-clarified pulp, the remainder was pellet. It is basically composed of fiber and seed remains, which would cause membranes fouling precociously.

Established permeate flux of clarified juice are reported in Table 1. In all tests the typical behavior of tangential microfiltration process curve was observed. After a sharp initial flux decline due to membrane consolidation and polarization, flux stabilization occurred at about 4 h for both membranes.

By observing Table 1 passion fruit pulp permeates flux decreased with the transmembrane pressure increase to the ceramic tubular membrane and increased not significantly to hollow fiber membrane. This is due to the fact that particles in suspension are forced to enter the membrane pores at higher pressures, causing flux decrease and membrane fouling or clog. Alvarez et al. (1996), Fukomoto et al. (1998), Oliveira et al. (2010), Vladisavljevic et al. (2003) and Chiampo and Conti (1999) relate similar behavior.

Constancy deviation between flux/permeate to high pressures is due to the polarized gel layer consolidation, which is formed in the passion fruit pulp because of the presence of pectin, once pectin has a high potential for forming gels. This layer ends up constituting a filtering path; it compacts with the transmembrane increase, resulting in the decrease of permeate fluxes.

In relation to the enzymatic treatment applied, it was observed that with the Pectinex Ultra SP-L concentration increase, there was a reduction on gel layer formation, since the enzyme has the function of degrading pectin, which enhances the reduction of permeate flux. In the tubular membrane, the degradation action was observed with the highest permeate flux reduction at the concentration of 150 ppm, and the lowest reduction at the concentration of 300 ppm. Observing the hollow fiber membrane, it was noticed that, at the concentration of 150 ppm, there was a small reduction in the flux, and when it was compared to the 300 ppm concentration, there was even an increase of permeate flux proving, thus, the enzyme effect on the destruction of the pectin molecule.

Comparing fluxes of both membranes, tubular membrane flux was significantly higher in comparison with hollow fiber membrane flux. It is due to the fact that they have different module recirculation flows, 500 L/h (Re = 10,800) for the tubular one and 325 L/h (Re = 250) for the hollow fiber one, in which the former has the maximum possible flow in the experimental module used. Several authors (Ushikubo et al., 2007; Vaillant et al., 1999; Vladisavljevic et al., 2003; Chiampo and Conti, 1999) affirm that cross-flow speed enhances the shear force on the membrane surface, avoiding particle deposition, minimizing the polarized layer, and resulting in a higher mass transference coefficient.

Having the higher permeate flux as a parameter to the best condition of operation, it can be stated that: (i) tubular membrane: transmembrane pressure of 0.5 bar, enzymatic concentration of 150 ppm; (ii) hollow fiber membrane: transmembrane pressure of 1.0 bar, enzymatic concentration of 300 ppm. For these best results, microbiological and sensorial analyses were carried out.

Membrane fouling may be expressed as a percentage reduction in permeability with water. Clogging percentage (CP) was determined by means of permeate flux, according to Eq. (9).

$$CP = 100\left[\frac{J_C - J_D}{J_C}\right] \quad (9)$$

Table 1
Passion fruit juice permeate flux in microfiltration.

Membrane	Tubular				Hollow fiber			
Enzyme concentration (ppm)	150		300		150		300	
Pressure (bar)	0.5	1.0	0.5	1.0	0.5	1.0	0.5	1.0
Permeate flux ($kg\ h^{-1}m^{-2}$)	42.4	26.9	40.0	39.0	18.3	18.7	17.4	19.5

Table 2
Membrane clogging percentage under different conditions.

Enzyme concentration (ppm)	150		300	
Pressure (bar)	0.5	1.0	0.5	1.0
Tubular membrane	54.3	85.1	51.3	84.3
Hollow fiber membrane	52.7	78.0	78.9	68.7

Here J_C is the water flux as feed through the clean membrane and J_D is the water flux through the fouled membrane. Clogging percentages determined by Eq. (9) considering the fluxes permeated with deionized water of the clean and the dirty membrane are presented in Table 2.

It can be noticed that the clogging percentage was kept from about 51% to about 85%, depending on the enzymatic treatment and transmembrane pressure applied. In general, as the transmembrane pressure increases, clogging percentage consequently increases too, except for the hollow fiber membrane with enzymatic treatment at 300 ppm.

4.2. Physico-chemical analysis

Physico-chemical analyses were performed for all runs. Table 3 shows physical and chemical properties of passion fruit juice under different enzymatic concentration. Rejection coefficients (RC) are presented in Tables 4 and 5 to ceramic and hollow fiber membranes, respectively.

As it can be noticed, soluble solids, color, absorbance and turbidity properties had a significant reduction, regarding the feed pulp and the obtained permeate, as it could be expected for a microfiltration process. It indicates that there was a concentration of the retained substance and the permeate clarification; it can be better visualized by observing the reduction coefficients, all positive.

Table 3
Physical and chemical properties of passion fruit juice after enzymatic treatment.

	Treated juice at 150 ppm	Treated juice at 300 ppm
pH (at 25 °C)	3.05	3.20
Color (APHA/Hazen/Pt-Co)	8250	8837
Absorbance	4.60	3.90
Turbidity (FAU)	1664	1386
Soluble solids (°Brix)	7.3	7.8
Total sugar (mg/mL)	75.5	56.2
Reducing sugar (mg/100 mL)	25.0	29.3
Galacturonic acid (mg/mL)	49.3	35.8
Vitamin C (μg/mL)	19.1	16.5

As for permeate total sugar level it was observed that, with the transmembrane pressure increase, sugar is significantly blocked from crossing the membrane resulting in a positive reduction coefficient. According to Cheryan and Alvarez (1995), this phenomenon happens due to the consolidation of the polarized gel layer, which is the filtering means, and compressed at high pressures resulting in the clogging percentage increase, and consequently in the reduction of permeate flux as well as in the coefficient rejection increase in these membranes. The enzymatic concentration increase caused this total sugar non-permeation effect to be higher. However, such diagnosis was not observed in the runs with tubular membrane at the enzymatic concentration of 150 ppm, in which it was verified a high sugar reduction in permeate at 0.5 bar of transmembrane pressure and a consequent decrease of sugar reduction with the transmembrane pressure elevation. In relation to reducing sugar, sugar increase restricted the passage of these compounds through the membrane, another indication of concentration polarization. Enzymatic concentration increase in the pulp had reverse effect, making it easier for the reducing sugar to cross the barrier, possibly for reducing the layer formed by the accumulation of pectinic substances.

Rejection coefficient for galacturonic acid increased with transmembrane pressure increase for tubular membrane, again evidencing the concentration polarization effect. The enzymatic treatment attenuated this rejection, once the enzyme acts by breaking these molecules of high molar mass. Nevertheless, for the hollow fiber membrane, the rejection coefficient decreased with the transmembrane pressure rise possibly due to the molecule affinity with membrane material. Such decrease was higher with enzymatic concentration increase.

Membrane rejection should not be attributed to vitamin C, for its molecule presents molar mass of 176 Da, therefore, could not be retained. Furthermore, they can easily be oxidized with light, oxygen and freezing time exposure, i.e., exposure to manipulation and general process. However, it is understood that the lower the rejection coefficient, the higher is the level of this attribute in the permeate, thus showing better quality. Thus, it can be observed that in tubular membrane, transmembrane pressure helped vitamin C to go through the membrane, while in the case of hollow fiber, transmembrane pressure did not interfere with permeation. Besides this, reduction coefficient was lower in hollow fiber membrane than in tubular membrane, causing the former to yield a better permeate.

4.3. Microbiological analysis

In order to carry out the sensorial analysis, a microbiological analysis of the products to be used in the sensorial test was carried out. The analysis included: counting of coliform bacteria; *salmonella* sp; mould and yeast; and standard counting in mesopohile dish. Only the two former ones are required under Brazilian legislation (BRASIL, 2001) for fruit pulps, concentrated or not, with or without thermal treatment, chilled or frozen.

Table 4
Rejection coefficients for physical and chemical analyses (ceramic membrane).

	pH	Color	Absorbance	Turbidity	Soluble solids	Total sugar	Reducing sugar	Galacturonic acid	Vitamin C
0.5 bar–150 ppm	0.04	0.97	0.93	1.00	0.14	0.49	0.15	0.79	0.26
1.0 bar–150 ppm	0.01	0.95	0.92	1.00	0.07	0.12	0.01	0.47	0.52
0.5 bar–300 ppm	0.02	0.95	0.89	1.00	0.17	0.23	0.39	0.82	0.03
1.0 bar–300 ppm	0.04	0.96	0.90	1.00	0.07	0.10	0.10	0.31	0.17

Table 5
Rejection coefficients for physical and chemical analyses (hollow fiber membrane).

	pH	Color	Absorbance	Turbidity	Soluble solids	Total sugar	Reducing sugar	Galacturonic acid	Vitamin C
0.5 bar–150 ppm	0.007	0.950	0.922	0.999	0.151	0.270	0.570	0.556	0.157
1.0 bar–150 ppm	0.000	0.947	0.920	1.000	0.083	0.118	0.124	0.540	0.065
0.5 bar–300 ppm	0.003	0.927	0.898	0.995	0.105	0.017	0.050	0.405	0.017
1.0 bar–300 ppm	0.006	0.976	0.927	1.000	0.151	0.620	0.162	0.386	0.130

Table 6
Microbiological analysis.

Product/Pulp	Mesophilic bacteria (UFC/mL)	Mould (UFC/mL)	Yeast (UFC/mL)	Total Coliforms (NMP/mL)	Coliforms at 45 °C (NMP/mL)	*Salmonella* spp. in 25 mL
Legislation	–	–	–	–	10^2	Absent
Pulp	5.4×10^2	$<1.0 \times 10^2$	$<1.0 \times 10^2$	<0.3	<0.3	Absent
Feed on tubular membrane	8.6×10^2	$<1.0 \times 10^2$	$<1.0 \times 10^2$	<0.3	<0.3	Absent
Permeate on tubular membrane	1.0×10^1	$<1.0 \times 10^2$	$<1.0 \times 10^2$	<0.3	<0.3	Absent
Feed on hollow fiber membrane	6.5×10^2	$<1.0 \times 10^2$	$<1.0 \times 10^2$	<0.3	<0.3	Absent
Permeate on hollow fiber membrane	4.0×10^2	1.4×10^3	$<1.0 \times 10^2$	<0.3	<0.3	Absent

In Table 6, it is possible to notice that the products attended the legislation specifications and thus are appropriate for consuming. Also Table 6 shows that the ceramic tubular membrane retained mesophilic bacteria, which allows us to conclude that it was efficient for cold pasteurization of the product. Meanwhile, hollow fiber membrane also retained these bacteria, but during the processing, there was a possible contamination, probably because this module is made of PVC and due to its shape, which is difficult for cleaning and sterilizing, subject to the formation of an internal layer of microorganisms.

As for mould, yeast, total coliforms and coliforms at 45 °C analyses, it was verified that, as presented in Table 6, the counting method employed could not quantitate accurately the microorganisms of each sample. It certified, though, that all products can be consumed without posing risks for the consumer's health, once they present minimal indexes.

4.4. Sensorial analysis

Results obtained from the variance analysis are presented in Table 7.

Table 7
Analysis of variance.

Juice	Mean[1]
Pulp (without microfiltration)	6.84[a]
Clarified by hollow fiber membrane	5.06[b]
Clarified by tubular membrane	6.31[a]

[1] Mean with the same letter does not differ at level of 95%, according to Tukey's test.

Then, comparing the means presented in Table 7, pulp juice and the hollow fiber membrane permeate juices were significantly different. For pulp juice and tubular membrane permeate juice there was no significant difference between their means. On the other hand, the permeate juices from tubular membrane and hollow fiber gave significant difference between the means.

4.5. Model fitting

In order to determine the flux as a function of time, the models listed in Eqs. (1)–(5) were fitted by Bayesian inference and parameter mean, standard deviation and DIC were obtained. DICs and estimated parameters for theses models as well as mean and standard deviation are presented in Tables 8 and 9.

Comparing DICs from Eqs. (1) and (2), models which describe flux as a function of time, a highly significant difference can be observed according to Eq. (8). DIC comparison between Eqs. (1) and (2) showed that Eq. (1) is a better fit than Eq. (2) due to low values. The parameter b_1, from Eq. (1), increased when transmembrane pressure was increased and enzyme concentration was kept constant, showing that it depends on transmembrane pressure. The same behavior was observed in the case of Eq. (2).

For models described by Eqs. (3)–(5), which describe pore blocking, cake filtration and internal pore blocking, it was observed through DIC analysis that internal pore blocking was more significant on ceramic membrane. This phenomenon, according to Barros et al. (2003), implies that the intrinsic membrane resistance changes during microfiltration by membrane fouling, which is due to either solute adsorption onto the membrane surface and membrane plugging. Fouling layer formation was controlled by

Table 8
Bayesian estimation model parameters for the microfiltration of passion fruit juice using ceramic membrane.

Bayesian estimations													
Pressure		0.5 bar						1.0 bar					
Enzyme concentration		150 ppm			300 ppm			150 ppm			300 ppm		
Model	Parameter	Mean	SD	DIC	Mean	SD	DIC	Mean	SD	DIC	Mean	SD	DIC
Eq. (1)	b_1	0.1007	6.91×10^{-3}	351	0.0664	2.92×10^{-3}	377	0.1263	2.60×10^{-3}	242	0.1422	1.98×10^{-3}	404
Eq. (2)	b_2	8.33×10^{-5}	1.28×10^{-5}	362	4.53×10^{-5}	2.59×10^{-6}	389	3.24×10^{-4}	9.16×10^{-5}	375	3.46×10^{-4}	9.59×10^{-5}	632
Eq. (3)	b_3	8.57×10^{-7}	1.33×10^{-7}	362	6.03×10^{-7}	3.42×10^{-8}	389	4.00×10^{-6}	1.15×10^{-6}	379	2.58×10^{-6}	6.82×10^{-7}	632
Eq. (4)	b_4	75.8	9.11	319	37.5	1.49	305	396.4	69.3	322	335.9	48.5	539
Eq. (5)	b_5	7.72×10^{-11}	1.00×10^{-10}	227	5.81×10^{-11}	1.00×10^{-10}	230	1.96×10^{-10}	1.05×10^{-10}	310	1.31×10^{-5}	1.01×10^{-10}	502

Table 9
Bayesian estimation model parameters for microfiltration of passion fruit juice using polyamide hollow fiber membrane.

Bayesian estimations													
Pressure		0.5 bar						1.0 bar					
Enzyme concentration		150 ppm			300 ppm			150 ppm			300 ppm		
Model	Parameter	Mean	SD	DIC	Mean	SD	DIC	Mean	SD	DIC	Mean	SD	DIC
Eq. (1)	b_1	0.130	9.69×10^{-3}	197	0.170	1.85×10^{-3}	706	0.111	3.94×10^{-3}	833	0.172	3.41×10^{-3}	751
Eq. (2)	b_2	4.48×10^{-5}	1.28×10^{-4}	218	1.94×10^{-3}	2.93×10^{-4}	1082	6.18×10^{-4}	4.47×10^{-5}	873	2.10×10^{-3}	1.80×10^{-4}	923
Eq. (3)	b_3	9.91×10^{-6}	2.06×10^{-6}	218	1.63×10^{-5}	2.32×10^{-6}	1082	9.51×10^{-6}	6.84×10^{-7}	873	1.93×10^{-5}	1.80×10^{-6}	923
Eq. (4)	b_4	641.6	107.2	179	3580	711.3	207	543.5	19.9	646	1251	51.4	720
Eq. (5)	b_5	5.12×10^{-10}	1.11×10^{-10}	274	5.74×10^{-9}	3.89×10^{-10}	782	5.74×10^{-9}	3.49×10^{-10}	782	6.95×10^{-9}	4.99×10^{-10}	843

how colloidal material specifically interacts with membrane surface. Riedl et al. (1998) affirms that the consolidation of fouling layer in rough membrane is slower than in smooth surfaces, allowing more solids to enter pores. Barros et al. (2003) say that the tortuous internal structure of ceramic membrane could also have produced more stagnant regions where solids could accumulate, leading to higher levels of pore plugging. These supposed rough membranes would have more stagnant zones on the membrane surface where turbulent flow would be unable to sweep away accumulated solids.

On the other hand, cake filtration was more significant on hollow fiber membrane. According to Todisco et al. (1996) this means that the convective flux direct from the bulk solution towards the membrane prevails on the rate of shear-induced back diffusion of the rejected material, leading to the formation of a cake on the membrane surface.

The pore blocking was more pronounced in ceramic membrane due to turbulent regime flow, which increases the rate transfer of the particles from the membrane to the main flow, resulting in a less compact cake. On the hollow fiber membrane molecules form cake, due to laminar flow, and are disposed to form aggregates with other macromolecules via hydrogen bonding.

5. Conclusion

From the tests carried out and results obtained in this work, it can be concluded that:

- Centrifugation, for removing materials in suspension, was important for reducing clogging in membranes and, consequently, for their best performance.
- The process efficiency was evaluated in terms of the rejection coefficient, and it was noticed that parameters such as pH, soluble solids, color, absorbance and turbidity behaved as expected in the case of membrane separation processes, in which the first parameter was practically unaltered, and the others had a high rejection, indicating that the permeate was clean and clarified.
- The best performance of ceramic tubular membrane was under 0.5 bar transmembrane pressure and 150 ppm enzymatic concentration. For hollow fiber membrane, the best conditions were: 1.0 bar transmembrane pressure and 300 ppm enzymatic concentration. Overall, the best performance, on the bases of flux values and physical and chemical analyses, was presented by the tubular membrane, for it showed low significant difference in comparison to the hollow fiber membrane.
- The microbiological analysis demonstrated that permeate comply with the current Brazilian legislation, showing the efficiency of the process at assuring clarification, as well as the pasteurization of the clarified pulp.
- Sensorial analysis confirmed that the hollow fiber membrane permeate had less acceptance when compared to pulp and tubular membrane permeate; the latter two products did not show any significant difference in acceptance between themselves.
- Mathematical models allowed the description of the fouling mechanisms involved in the membrane process. In the case of the ceramic tubular membrane, internal pore blocking predominates, whereas, cake filtration dominated in the case of hollow fiber membrane.

Acknowledgement

The authors would like to acknowledge CNPq and CAPES for financial support.

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